A STUDY OF THE ATMOSPHERE AS A DYNAMIC PATHWAY FOR THE ACCUMULATION OF CROP APPLIED PESTICIDES

by

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A study of the atmosphere as a dynamic pathway for the accumulation of crop applied pesticides

by

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INTRODUCTION

Pesticides are used on a large scale on the Canadian prairies. On the average 5×10^5 kilograms of phenoxy herbicides are used annually on cereal crops, for the control of broad leaf weeds. This represents about 30 million acres of surface applied chemical.

It is well recognized that the major route for the transport of pesticides from the soil or water surfaces to the atmosphere is by vapour loss, both during and following their application.

Cole (1977) reported on a series of laboratory experiments whereby he attempted to identify the primary relocation sites of selected pesticides which were applied to soil or crop. The transfer of tagged radioactive residues to all three major environmental compartments, land, water and air was considered.

The pesticide dieldrin was studied in the model ecosystem. Dieldrin was injected into the soil at the time of planting. At termination of the project the fractions of the pesticide residue in each segment of the ecosystem was determined. Dieldrin fractionation was somewhat sensitive to the soil substrate type; with vermiculite sediment, 43%, 5% and 47% of the pesticide were found in soil, animals and air, whereas for soil sediment 37%, 1.2% and 61% of the pesticide were found in the same respective groupings.

Since it can be shown that certain pesticides readily enter the atmosphere by volatilization of a surface residue, it is only obvious to ask where and how much of that residue will eventually return to the surface and in what form. The fact that many pesticides are now being

found in remote environments, such as the open ocean (Harvey, 1974), leads only to the conclusion that the atmosphere is a direct and quantifiable source of environmental pollution for the entire ecosystem.

On the Canadian prairies Grover et al (1976) has studied the levels of pesticide in the air throughout the growing season. Up to 50% of the daily samples collected at any location during the year contained pesticide (2,4-D), the mean daily atmospheric concentrations being in the range from 0.01 to 13.50 $\mu g/m^3$. Recently, detectable amounts of pesticide, such as triallate, have been observed on the prairies on almost any growing day of the season (Grover et al, 1981).

By using techniques of micrometeorology it is possible to calculate fluxes from measured pesticide concentration vapour profiles. The approach has been summarized by Taylor (1978) and it represents a significant improvement to the total mass balance of pesticides in the environment. Prior to this approach pesticide mass balance to the atmosphere was calculated by difference from other systems. With this new approach the amount of pesticide entering the atmosphere was calculated directly. However, knowledge of the turbulence structure of the atmosphere's surface flux layer is required.

The vertical flux of pesticide vapour (F_t) through a horizontal plane at height z above the crop canopy is given by the equation:

$$F_t = K_z (dc/dz)$$ (Parmele et al, 1972).

where dc/d is the gradient of vapour concentration with height and K_z is the vertical eddy diffusivity coefficient at height z. The value K_z depends upon the turbulent flow into which the vapour is dissipated. It is related to the meteorological conditions and not to any physical

or chemical property of the pesticide. A portable micrometeorological tower has been designed to monitor the variations in the eddy diffusivity coefficient (Shewchuk and Grover, 1981).

Pesticide concentrations in the air above the crop or soil surfaces are measured by drawing air at a known flow rate through a sampling device containing a solid absorbing medium. Polyurethane foam has been used successfully to measure concentrations in air at levels well below 1 mg/m^3 according to Turner (1977). Six levels of air sampler are required in order to establish an adequate gradient. Each series of samples was changed every two or four hours to be compatible with the micrometeorology data.

PROCEDURE

The experiment designed under this contract was conducted at Canada Agriculture, Regina. Many of the details of field preparation, seeding and spraying methodologies are given in Grover et al, 1982.

The experiment began at 10:30 local standard time on June 25, 1981. It continued for approximately one month. The primary determinant for experiment duration, however, was the presence of pesticide mass flux.

RESULTS

A weather data and conditions summary is shown in Table 1. Except for a period when 3.8 mm of rainfall was recorded on day three, the period of observation was characterized by clear and cloudless skies. Pesticide mass flux as determined from data collected is seen in Figure 1. All calculations which were used to derive this figure are given by a procedure outlined in Shewchuk and Grover (1981).

As seen in the figure, the pesticide mass flux was greatly reduced after the second day. Thus, the calculations of flux to the atmosphere were terminated after the fifth day. A rain shower which occurred in the early morning of the third day is seen as a peak in the figure.

Total mass of pesticide volatilizing to the atmosphere is 290 grams. This represents approximately 16% of the applied chemical. A draft paper outlining the fate of this pesticide to other sectors of the ecosystem is shown in the appendix.

Table 1. Weather conditions during the first week of the study [from Grover et al 1983 (in press)].

Date	Sample Day	Rainfall[1]	Wind[2]	Temperature Air[3] Max.	Air[3] Min.	Soil[4]	Soil Moisture[5] 0500	Soil Moisture[5] 1500	General Weather Conditions
		mm	m/s	------- °C ------			%		
25 June	1	--	2.6	23.6	8.5	13.5	16.7	6.8	Clear
26 June	2	--	4.9	28.8	8.4	14.0	12.3	5.8	Clear
27 June	3	3.8	3.9	22.7	10.2	17.0	41.2	14.0	Clear
28 June	4	--	2.7	20.3	7.8	15.5	15.1	8.8	Cloudy, 30%
29 June	5	--	3.6	24.4	4.2	14.5	13.0	6.0	Clear
30 June	6	--	4.0	29.8	11.6	17.0	9.4	-	Clear
01 July	7	--	3.4	27.3	12.8	19.0	-	-	Cloudy, 10%

[1]Major rainfall events (10 mm +) during the 35-day study period were day numbers 18, 23, and 35 which received 42.6, 12, and 11 mm of rainfall, respectively.

[2]Mean wind speed at 1 m height.

[3]At 1 m height.

[4]Mean temperature at 5 cm depth.

[5]Surface 1 mm samples taken daily at 0500 and 1500 hours.

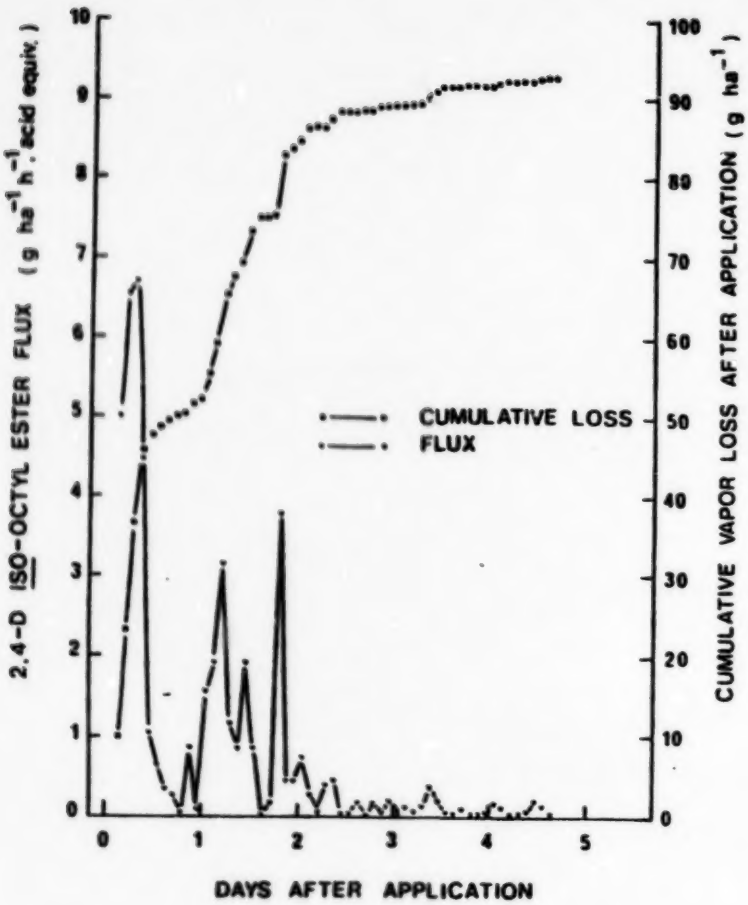

Figure 1. Vapour flux of pesticide into the atmosphere as a function of time after application from [Grover et al 1983 (in press)].

DISCUSSION

The pesticide mass flux into the atmosphere was successfully determined with this study. It is during the first day of the experiment that volatilization rates of chemical to the atmosphere are the highest. Subsequently, the decay for successive days proceeds quickly and by the fifth day little mass flux is seen. This indicates that the pesticide is being hydrolyzed to an acid form.

Because of the fact that mass fluxes of pesticide as determined by this technique depend only on the eddy diffusivity coefficient and the gradient of air concentration, the method outlined is applicable to determine the mass fluxes of any pesticide from the soil or crop. The eddy diffusivity coefficient is determined exclusively from the turbulence structure of the atmosphere over which the pesticide trials are conducted and the gradient of pesticide concentration is determined by whatever means are applicable to trap the pesticide of interest from the air (in this case the polyurethane foam plug was used).

In order to understand the behavior of the atmosphere as a dynamic pathway for the accumulation of crop applied pesticides, the principles of micrometeorology turbulence must be applied to diffusion of organic vapour molecules from the surface. The approaches outlined in this contract are acceptable procedures for the determination of mass fluxes of pesticides in general.

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APPENDIX

Draft paper of the "Fate of 2,4-D iso-octyl ester after application to a wheat field" by R. Grover, S.R. Shewchuk, A.J. Cassna, A.E. Smith and J.H. Hunter.

Fate of 2,4-D iso-octyl Ester after Application to a Wheat Field

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ABSTRACT

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INTRODUCTION

Monitoring of airborne residues, both in the Canadian prairies and elsewhere, has shown that the post-application volatilization losses of herbicides plays an important role in their dissipation in the environment (Farwell et al. 1976, Grover et al. 1976, 1981). More recently, however, the significance of volatilization losses has been demonstrated on a quantitative basis for trifluralin and EPTC, where post-application losses by vapor activity were reported to be about 25% and 75%, respectively (White et al. 1977, Cliath et al. 1980). Quantitative documentation of actual losses under typical field conditions, where estimates were made of total volatilization losses along with herbicide dissipation in the crop and soil components, are non-existent. This paper reports the magnitude of 2,4-D iso-octyl ester losses by volatilization after application to a wheat field along with the relative partitioning of the ester into the soil and crop components in the field and its subsequent dissipation from each of these compartments.

MATERIALS AND METHODS

Crop Seeding and Herbicide Application

The study area, located on the north-eastern part of the Research Station, Agriculture Canada, Regina, had over-wintered as standing stubble during the winter of 1979/1980. In 1980, the field was summer-fallowed during which period it was disced, cultivated twice, and also rod-weeded. The top 5 cm of the Ap horizon of the soil (% sand, % silt, % clay) had % organic matter and a pH of . On May 1, 1981, 3.7 ha of the field was seeded to wheat (Triticum aestivum L. var. Neepawa) at 100 kg/ha with a discer set at a depth of 7 cm. No fertilizer was added. The wheat seedlings emerged on May 14. On June 25, when the wheat seedlings were 20 cm in height and had five leaves and two tillers, the field was sprayed with the iso-octyl ester of 2,4-D (2,4-dichlorophenoxyacetic acid) at a rate of 0.5 kg/ha (acid equivalent) between 0900 and 1000 h (CST), using a tractor-mounted sprayer. The sprayer boom, 7.4 m wide and fitted with 80° nozzles, was set at 58 cm above the ground to give a uniform coverage on the target plants at a 15 cm height. The sprayer was calibrated to deliver 100 liters/ha of 2,4-D emulsion at 207 kPa while travelling at 5 km/h.

Air Sampling

Air samples were collected downwind during application at a temporary sampling site (0900 to 1000 CST). Immediately after application, all sampling and meteorological equipment was installed in the center of the field and subsequent sampling started at 1030 CST. Air was sampled continuously for the next six days, using 2-h sampling interval. The location of the sampling mast in the center of the field ensured an adequate fetch length (about 100:1) for profile measurement. Air sampling system similar to that reported by Cliath et al. (1980) was employed

in monitoring 2,4-D ester concentrations in the air. Samples were collected at the 30, 50, 75, 100, 150, and 200 cm heights above the crop canopy. Each of the vapor traps, a 45 by 50 mm polyurethane plug, was aspirated at 25 liters/min. The trapping and extraction efficiency of the foam plug for 2,4-D iso-octyl ester have been reported earlier (Grover & Kerr, 1981).

Flux calculations

Pesticide flux calculations were carried out by two separate methods, the aerodynamic and energy balance methods (Parmele et al., 1972). The flux is determined as a product of the K-value and the gradient of pesticide concentration (Shewchuk and Grover, 1981).

Profiles of wind, dry-bulb temperature and wet-bulb temperature were determined in the atmosphere with a six level micrometeorological system (Shewchuk and Grover, 1981). The height of the levels was determined starting at the top of the crop canopy. The levels were spaced at intervals similar to the air samplers. The net radiation (Fritschen, 1965) was determined at the one meter level and soil heat flux was measured with a heat flux plate. The heat flux plate was placed several millimeters below the soil surface. In addition a profile of soil temperature was determined for depths of 0.02, 0.05 and 0.10 m. A tipping bucket rain gauge and a potentiometric wind vane were used for rainfall amounts and wind direction, respectively.

Wind sensors were a three cup rotating miniature anemometers (Bradley, 1969) fitted with a light-chopper type pulse generator and power amplifier circuit. The sensors of dry-bulb, wet-bulb and soils temperature were thermistor networks. The wet and dry-bulb sensors were placed in a radiation shield and aspirated at 3 m/s.

All micrometeorological data were collected at 15 min intervals by a field positioned data acquisition system. Eddy diffusivity coefficients were calculated by a computer program for fluxes for a 2 hour sampling period. This period was used to coincide with the times of collection for the pesticide samples. All profiles were corrected for the effects of stability. With the aerodynamic method the zero plane displacement parameter was usually fixed to be 0.7 times the height of the canopy (Montieth, 1973). The KEYPS (Sellers, 1969) function was the principle correction applied to the profiles. The calculation of eddy diffusivity coefficient is based on the assumption that the eddy diffusivity coefficient for sensible heat, latent heat and pesticide flux are similar.

The location of the towers was in the middle of the field and all were placed within a 10 meter diameter circular plot. Field data are initially placed on ferric oxide tape cassette and later transferred to a laboratory facility for calculations and analysis.

Crop Sampling

The wheat field was divided into four approximately equal sized quadrants for crop and soil sampling. Two composite samples of green wheat tissue were collected from each quadrant at the following times: before spraying, within 1 h after spraying on day 1 and on 2, 4, 8, 18 and 34 days after spraying. A composite sample consisted of 10 subsamples collected at random within a quadrant. Each subsample was cut at the soil surface from a 4 ha (20 cm X 20 cm) area. One set of composite samples was analyzed immediately after collection for 2,4-D iso-octyl ester residues, whereas the other set, reserved for total 2,4-D acid analysis, was chopped, using a food cutter, placed in polyethylene freezer bags and stored at -10°C until extraction.

A sample washer was constructed from 18 gauge flat mesh expanded metal (8 mm) and was 260 mm X 70 mm i.d. cylinder. The bottom was attached to the cylinder with three pieces of copper wire. After the washing operation was complete, the bottom was removed and the wheat tissue removed from the cylinder by pushing it out through the bottom end. The handle of the sample washer was 360 mm in length.

Soil Sampling

A check soil sample was taken one day prior to the spraying with 2,4-D, while soil samples were taken immediately after spraying on day 1 and on 2, 4, 8, 18 and 33 days following crop spraying. From each quadrant of the field, six soil cores 7.5 cm in diameter and 7.5 cm in depth (total surface area of 265 cm^2) were randomly sampled. The samples taken from each quadrant were pooled and separately bagged in plastic sacks. Immediately after collection, the soil samples were taken to the laboratory and weighed (approximately 2 kg) and poured through a Riffle Sampler to divide the soil into two approximately equal parts. Each part was then separately and further divided by repouring through the Riffle Sampler until there was about 100 g soil remaining. From each of the two reduced samples, representing duplicate subsamples from each of the field quadrants, 20 g portions were weighed into 125-ml glass-stoppered flasks which were frozen at -10°C to await extraction and analysis. A moisture determination was carried out on the remaining soil from which the samples had been taken by heating 50 g of the field soils at 100°C for 24 h.

Analytical Procedures

Extraction and analysis of air samples : Airborne residues of 2,4-D iso-octyl ester were soxhlet extracted and analyzed gas chromatographically using a procedure described earlier (Grover and Kerr 1981).

Extraction, cleanup and analysis of ester in the crop : Each composite sample was

weighed prior to analysis. All of the sample was then inserted into the cylindrical sample washer (Figure 1) and immersed with occasional agitation in 1000 ml methylene chloride in a 2-L graduated cylinder for a period of 5 min. The sample washer was then raised above the methylene chloride solution and the methylene chloride allowed to drain from the plant tissue. The plant tissue, while still in the sample washer, was rinsed with sufficient methylene chloride to return the volume in the graduated cylinder to 1000 ml. Fifty milliliters of the leaf surface wash was then transferred to 100 ml RB flask and taken just to dryness using a rotary evaporator.

At later harvest dates, the sample holder was not large enough to hold an entire composite sample. The composite samples were then divided approximately in half, and each half was washed as described above, except that the second half was washed in the same 1000 ml methylene chloride as the first half of the sample.

Florisil (4. ml, deactivated with 5% water, Fisher Scientific) was added to 10 ml hexane in a 10 mm i.d. X 200 mm column, topped with 1 cm anhydrous sodium sulfate, and the hexane drained to the surface of the sodium sulfate. The residue on the 100 ml RB flask was transferred to the Florisil cleanup column with two 1.5 ml hexane rinses of the RB flask. The column was then eluted with 27 ml 0.5% acetone in hexane, the last 12 ml of which were collected. The eluate from the column was then concentrated to approximately 0.5 ml using a rotary evaporator and then taken to volume (1 ml) with hexane for gas chromatographic analysis.

Extraction, cleanup and analysis of total acid in the crop : The green wheat tissue was extracted as described previously (Cessna, 1980) with the following changes: only 25 g green tissue was extracted, the alkaline extract was taken to volume (250 ml) and the succeeding workup was carried out with 50 ml alkaline

extract (equiv to 5 g green tissue).

The combined chloroform extracts were concentrated just to dryness using a rotary evaporator and the extract residue was transferred to a 20 mm i.d. X 100 mm test tube (19/26 joint) with two 1.5 ml methanol rinses of the 100 ml RB flask and then 3 ml BF_3/methanol reagent (14% by weight) was added. The test tube was tightly stoppered and heated for 30 min at 70C (wax bath). The reaction mixture was cooled (ice-water bath), 10 ml cold 5% Na_2SO_4 solution added, and the mixture extracted three times with 3 ml hexane, the hexane layer being transferred to a Florisil cleanup column after each extraction.

Extraction and analysis of ester and acid in the soil : All analyses were carried out within four days of collection by shaking each soil sample on a wrist-action shaker for 1 h with sufficient 20% aqueous acetonitrile containing 2.5% of glacial acetic acid, so that the combined volume of extractant together with the water present in the soil was equivalent to 50 ml. After centrifugation at 3500 rpm for 4 min, 25 ml of supernatant were shaken with 100 ml of 5% aqueous sodium carbonate solution and 25 ml n-hexane in a 250 ml separatory funnel. After settling the organic layer was examined gas chromatographically for iso-octyl ester of 2,4-D.

Recoveries of 2,4-D iso-octyl ester from 4 replicate samples of air-dried heavy clay, fortified at the 1.0 g/g level for 24 h prior to extraction, were all in excess of 95%. This indicated that no significant hydrolysis of the ester to the acid was occurring during extraction. Using aqueous acidic acetonitrile as extractant, the recovery of 2,4-D acid from the heavy clay is 90% (Smith, 1978). Thus no recovery corrections were applied to any of the analytical data.

Following analysis, the concentrations of both 2,4-D octyl ester (as acid equivalent) and 2,4-D acid, per duplicate soil sample, were calculated and

averaged. Hence, the amounts of 2,4-D per pooled quadrant soil was calculated. Since the weight of this soil was equivalent to 273.6 cm^2, amounts of 2,4-D remaining as either ester or acid could be determined as kg/ha. At each sampling date the mean concentrations of acid and ester forms of 2,4-D remaining were calculated by averaging the data obtained from the four quadrants.

Analysis of the check field soils taken 24 h prior to spraying showed that they contained less than 0.01 kg/ha of either the iso-octyl ester of 2,4-D (as acid equivalent) or of 2,4-D acid.

Gas chromatographic analysis

The gas chromatographs used were Tracor 560 or Hewlett-Packard 5700/5730A Models, all equipped with ^{63}Ni electron-capture detectors. The glass columns (1.8 or 1.5 m by 4 mm i.d.) were packed with 100 to 120 mesh Ultrabond-20M. Carrier gas was argon containing 5% methane, at flow rates of 30 to 40 ml/min. Depending on the carrier gas flow rate and the column temperature conditions, the retention times for the iso-octyl ester were 3.77 or 4.55 min and thatt for the methyl ester 3.5 and 3.8 min.

RESULTS AND DISCUSSION

General weather conditions, including the soil moisture and soil temperature conditions, during the week following the application of 2,4-D ester are shown in Table 1. The week was, in general, a dry period, except for a brief shower between 0100 and 0130 h on day 3. The dry conditions continued through to the second week, with the first major rainfall event on day 18 when the field received 42.8 mm of precipitation. The wide fluctuations in the daily maximum and minimum air temperaturs were typical for this area. The surface soil moisture levels also showed a diurnal trend, with higher levels in the early part of the morning and the lower levels in the afternoon, the former no doubt due to dew deposition. It may be pointed out that the surface soil moisture conditions were, in general, near or even below the wilting point level, except after an rainfall event.

CONCLUSIONS

1. Over 99% of the amount applied was deposited on the target.
2. Post-application losses (off-target) of the ester were primarily to the air, about 20% being lost as vapor within 48 hours after application.
3. Major portion of the vapor losses occurred from the deposits on the crop and these losses were temperature dependent.
4. Ester losses from the soil as vapor occurred only when the soil surface was moist, i.e. after a rainfall event, or during early hours of the morning due to deposition of dew.
5. Hydrolysis to the acid form in the soil, together with subsequent degradation of the acid, was dependent on the availability of soil moisture.
6. On entry into the crop, the ester appeared to be hydrolyzed and the acid was either rapidly metabolized or conjugated; once conjugated, subsequent dissipation appeared to be primarily by dilution as the crop grew.

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LEGENDS FOR FIGURES

Fig. 1 Concentrations of 2,4-D iso-octyl ester in air (75 cm height), in soil (0-7.5 cm depth), and in crop over 8 days after application.

Fig. 2 Volatilization rate and cumulative losses of 2,4-D iso-octyl ester over 5 days after application.

Fig. 3 Amounts of total 2,4-D (ester and acid) and acid alone in wheat over a 34-day period in the field.

Fig. 4 Amounts of total 2,4-D (ester and acid) and acid alone in the top 0-7.5 cm soil layer over a 33-day period in the field.

Table 1. Climatic data and weather conditions during the first week of the study.

Date	Sample Day	Rainfall[1]	Wind[2]	Temperature			Soil Moisture[5]		General Conditions
				Air[3]		Soil[4]			
				Max.	Min.		0500	1500	
		mm	m/s	--------- °C ---------			%		
25 June	1	--	2.6	23.6	8.5	13.5	16.7	6.8	Clear
26 June	2	--	4.9	28.8	8.4	14.0	12.3	5.8	Clear
27 June	3	3.8	3.9	22.7	10.2	17.0	41.2	14.0	Clear
28 June	4	--	2.7	20.3	7.8	15.5	15.1	8.8	Cloudy, 30%
29 June	5	--	3.6	24.4	4.2	14.5	13.0	6.0	Clear
30 June	6	--	4.0	29.8	11.6	17.0	9.4	-	Clear
01 July	7	--	3.4	27.3	12.8	19.0	-	-	Cloudy, 10%

[1]Major rainfall events (10 mm +) during the 35-day study period were day numbers 18, 23, and 35 which received 42.6, 12, and 11 mm of rainfall, respectively.

[2]Mean wind speed at 1 m height.

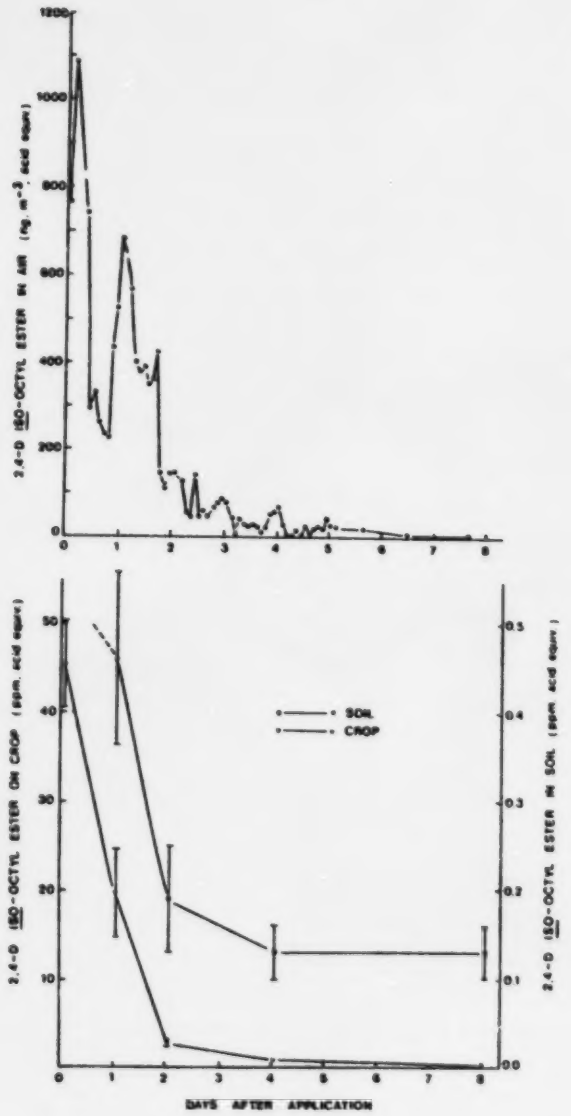
[3]At 1 m height.

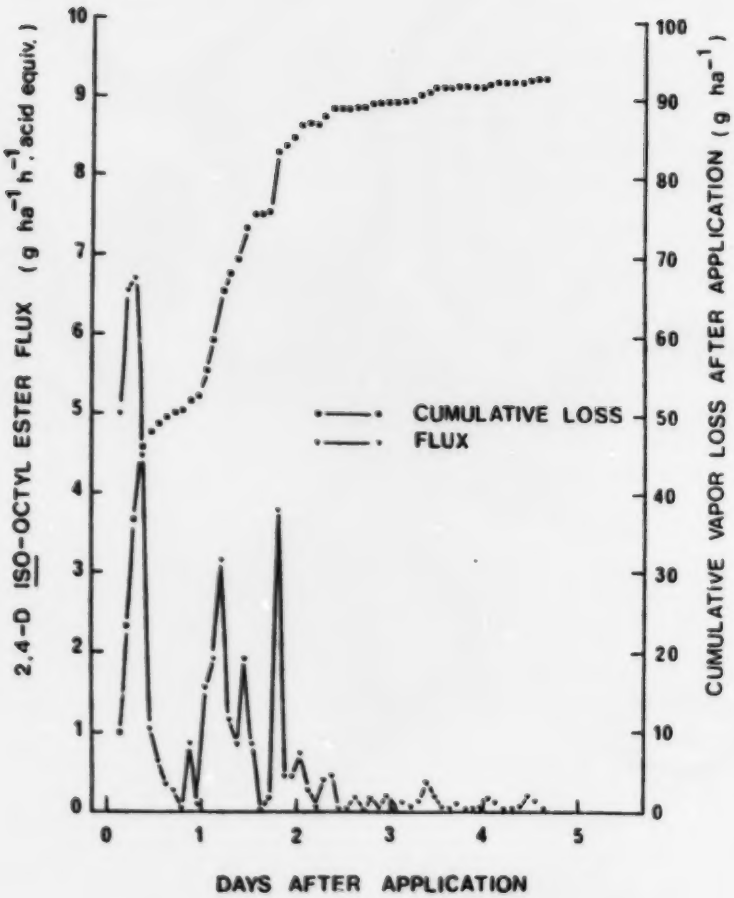
[4]Mean temperature at 5 cm depth.

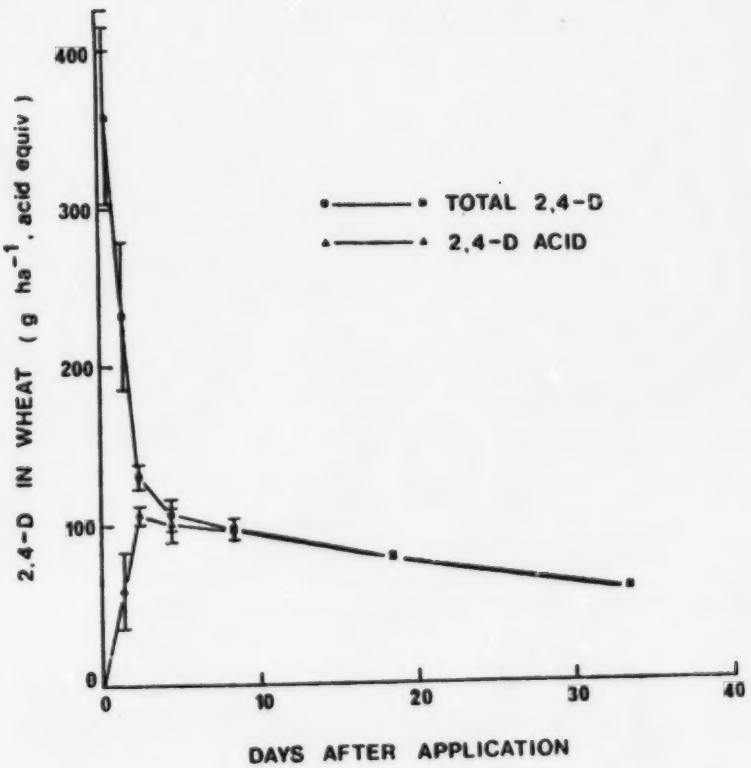
[5]Surface 1 mm samples taken daily at 0500 and 1500 hours.

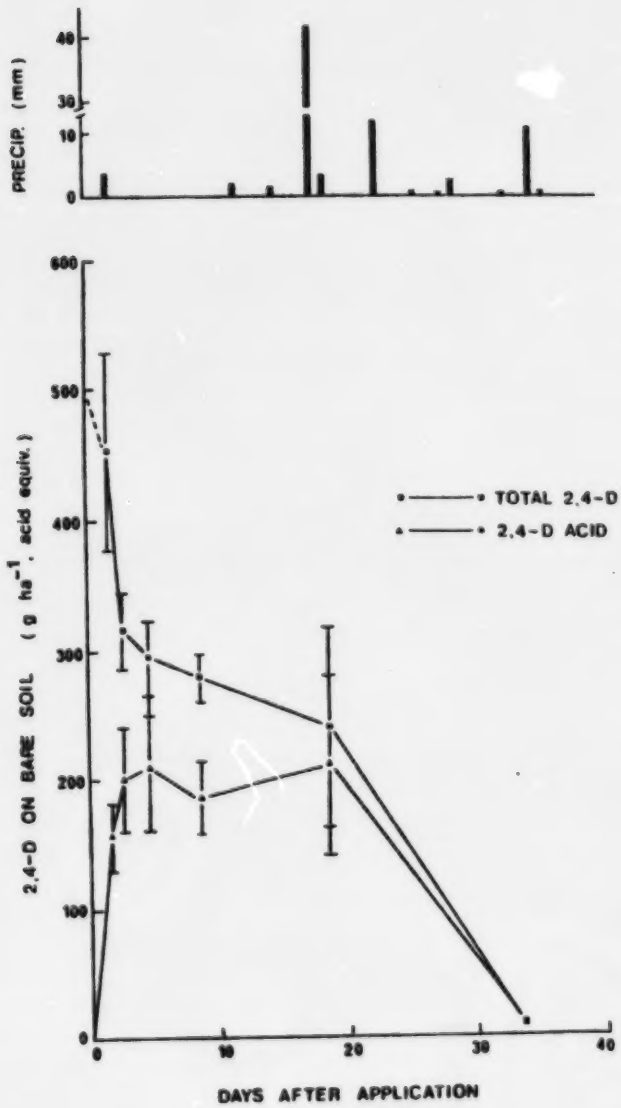
Table 2. Cumulative losses of 2,4-D iso-octyl ester from volatilization

Time, day	Cumulative amount volatilized as % of total	
	applied	volatilized
Application	0.2	0.9
1	14.5	60.0
2	21.0	91.4
3	22.1	96.6
4	22.5	98.7
5	22.6	99.9


2,4-D ISO-OCTYL ESTER IN AIR (ng. m⁻³ acid equiv.)
2,4-D ISO-OCTYL ESTER ON CROP (ppm. acid equiv.)
2,4-D ISO-OCTYL ESTER IN SOIL (ppm acid equiv.)
SOIL
CROP
DAYS AFTER APPLICATION


2,4-D ISO-OCTYL ESTER FLUX (g ha^{-1} h^{-1}, acid equiv.)
CUMULATIVE VAPOR LOSS AFTER APPLICATION (g ha^{-1})
CUMULATIVE LOSS
FLUX
DAYS AFTER APPLICATION
0
1
2
3
4
5
10
20
30
40
50
60
70
80
90
100


2,4-D IN WHEAT (g ha^{-1}, acid equiv)
400
300
200
100
0
0
10
20
30
40
DAYS AFTER APPLICATION
TOTAL 2,4-D
2,4-D ACID


PRECIP. (mm)
40
30
10
0
600
500
400
300
200
100
0
2,4-D ON BARE SOIL (g ha⁻¹, acid equiv.)
TOTAL 2,4-D
2,4-D ACID
0
10
20
30
40
DAYS AFTER APPLICATION